

IN THE JAPANESE PATENT OFFICE

TRIAL NO. 26872/95

and

TRIAL NO. 26875/96

AFFIDAVIT OF DR. GENE DRESSSELHAUS



Gene Dresselhaus, being duly sworn deposes and says:

1. I reside at 147 Jason Street, Arlington, Massachusetts USA 02174.
2. I hold a degree of BA from University of California, Berkeley and hold the Ph.D. degree from University of California, Berkeley.
3. I am Senior Scientist at the Francis Bitter Magnet Laboratory at Massachusetts Institute of Technology, Cambridge, Massachusetts.
4. I have been involved as a professor and researcher in the field of graphite, carbon fibers, and carbon nanotubes since 1965. I am the author with M.S. Dresselhaus and P.C. Eklund of "Science of Fullerenes and Carbon Nanotubes", Academic Press (1996). I am also the author with M.S. Dresselhaus, K. Sugihara, I.L. Spain and H.A. Goldberg of "Graphite Fibers and Filaments", Springer-Verlag (1988).
5. I make this affidavit in defense of Japanese Patent Nos. 1,701,869 and 1,869,277.
6. Vapor grown carbon fibers (a term usually limited to catalytically grown fibers) are an important class of fibers for scientific and industrial proposes. A subclass of such fibers are those comprised of carbon atoms in planes (graphene planes) forming cylinders and oriented parallel to the fiber axis. These have the highest elastic moduli and mechanical strength.
7. Such fibers are often described as having an "onion skin" or "tree ring" morphology. They include a central

hollow core, an initial growth region of ordered carbon and an outer thickened sheath of pyrolytic carbon.

8. In the growth process a thin hollow tube is first formed from a metal catalytic particle. The tube is thickened by deposition of carbon on its surface. In this thickening process, the tube acts as a nucleation site for the thermal dehydrogenation of gaseous hydrocarbons.

9. Vapor grown carbon fibers are often heat treated at temperatures of up to 3000°C. They may be converted in such processes to a more ordered turbostratic tree-ring coaxial cylinder morphology. In the case of larger diameter fibers, faceted fibers having a polygonal (non-circular) cross-section are formed. These faceted fibers are closest to crystalline graphite in both their crystal structure and properties.

10. The high temperature treatment process is referred to generally as annealing or sometimes as "graphitization". Disordered carbon e.g. pyrolytically deposited carbon may be converted under annealing or graphitization conditions to turbostratic, i.e. carbon graphene planes (not cylinders) which are parallel to the axis of a fiber.

11. The ability of annealing or graphitization processes to change the arrangement of carbon atoms in a fiber is limited by thermodynamic considerations. Annealing or graphitization is effective to correct lattice defects in graphene layers or to align slightly misoriented domains adjacent to one another. As a general proposition, major structural rearrangements are not possible. For example, fibers having a

fishbone orientation cannot be converted to those having graphene layers parallel to the axis of the fiber because too much energy is required to break and remake all of the bonds.

12. In my opinion, if a fibril is not grown as a structure that approximates coaxial cylinders, then no amount of heat treatment will convert the fibril into such a structure. The suggestion that almost any heat-treated fiber will be "identical" to the patented fibrils is not based upon scientific principles and is therefore without merit.

13. Where pyrolytically deposited, disordered carbon is converted to graphenic layers, these may assume a growth ring-like configuration where the fiber is relatively thin but may assume a polygonal configuration where the fiber is larger in diameter.

14. In my experience, annealing or graphitization processes are not effective to straighten fibers having sharp bends or kinks.

15. I am acquainted with the work of Professor Morinobu Endo of Shinshu University. I have collaborated with Professor Endo since 1982 in investigating various carbon fibers and nanotubes and I am familiar with his publications.

16. In this regard, I have been shown a copy of the publication "Industrial Materials, Vol. 30, pp. 109-115 (1982). My attention has been directed specifically to Photograph 8 in this work by Professor Endo and others.

17. From the photograph and the text relating to it, I am unable to determine the arrangement of the carbon atoms in the

fiber shown. This fiber appears to be among the class of "kinky" fibers discussed in par. 14 above which cannot be converted to graphene layers parallel to the fiber axis by heat treatment at high temperature, e.g. 2500 - 3000°C. Thus, it is my considered opinion that the serpentine and kinky fiber depicted in Photograph 8 would not become either straightened or uniform in cross-section if it were treated at high temperatures.

Gene Dresselhaus
GENE DRESSELHAUS

Sworn to before me this
10th day of October, 1996

Shawn L. Blum
Notary Public

My Commission Expires December 12, 1997.

